

DEPARTMENT OF COMMERCE

MAY 1 1925

BUREAU OF STANDARDS

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[Issued April 1, 1925]

RECOMMENDED SPECIFICATION FOR LIMESTONE,
QUICKLIME, LIME POWDER, AND HYDRATED LIME
FOR USE IN THE MANUFACTURE OF SUGAR

[This is the ninth of a series of specifications for lime that is used in various chemical industries. To assist in the development of these specifications the bureau has called together an Interdepartmental Conference on Chemical Lime, composed of representatives of the Geological Survey and Bureau of Mines, of the Interior Department; the Bureau of Soils, Bureau of Chemistry, Forest Service, and Fixed Nitrogen Laboratory of the Department of Agriculture; and the Chemical Warfare Service, of the War Department. The present specification, based on a draft originally prepared by M. J. Proffitt, of the polarimetry section, Bureau of Standards, has been unanimously approved by the above conference, by the National Lime Association, and by the Hawaiian Sugar Planters Association.]

ABSTRACT

Lime is used in the sugar industry either to precipitate impurities from the juices or sirups or, in the Steffen process, to precipitate the sugar from impure solutions. For the former purpose either lump quicklime or hydrated lime may be used; for the latter purpose very finely ground quicklime, known as lime powder, is required. For the purification of juices 85 per cent purity is required of the lime or hydrate or of the limestone from which they are made. When used in the Steffen process, lime powder must contain 90 per cent sugar soluble lime, and the quicklime or limestone used in making it must be correspondingly pure.

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I. GENERAL

1. USE OF LIME IN THE SUGAR INDUSTRY

(a) **USE OF LIME PRODUCTS.**—Lime is used in the sugar industry either to precipitate impurities from the juices or sirups or, in the Steffen process, to precipitate the sugar from impure solutions. For the former purpose either lump quicklime or hydrated lime may be used; for the latter purpose very finely ground quicklime, known as lime powder, is required. When the process requires the addition to the juice of an excess of lime which is afterward precipitated by carbon dioxide, the sugar manufacturer usually finds it economical to buy lime and carbon dioxide together in the form of limestone.

(b) **KIND OF PRODUCTS REQUIRED.**—This industry requires the purest grade of high calcium lime products which can be economically obtained. Lime powder in particular must be exceptionally pure as well as finely ground.

2. LIMESTONE

(a) **DEFINITION.**—Limestone used in the manufacture of sugar consists essentially of calcium carbonate.

(b) **USE OF LIMESTONE.**—Limestone is calcined to carbon dioxide and quicklime in a suitable kiln. The size of the stone depends upon the type of kiln. For a shaft kiln the stone should be uniform in size to permit uniform calcination and not too fine, lest the draft be choked. An exceptionally pure grade of stone is required if lime powder is to be made.

3. QUICKLIME

(a) **DEFINITION.**—Quicklime as used in the sugar industry is the product resulting from the calcination of limestone and consists essentially of calcium oxide. It will slake when water is added to it, and this slaking is accompanied by an evolution of heat and an increase in volume.

(b) **USE OF QUICKLIME.**—Quicklime can be slaked to form milk of lime, which is used to precipitate impurities from the juices, or it can be ground to form lime powder for use in the Steffen process. Pure high calcium lime is required in either case.

4. LIME POWDER

(a) **DEFINITION.**—Lime powder is finely divided quicklime.

(b) **USE OF LIME POWDER.**—Lime powder is added to cold diluted molasses in the Steffen process to precipitate calcium saccharate, leaving the impurities in solution. For this purpose it must be properly calcined, very fine, and exceptionally pure. Even air-slaking renders lime inactive for this purpose.

5. HYDRATED LIME

(a) **DEFINITION.**—Hydrated lime is a dry powder which is made by treating quicklime with enough water to satisfy its chemical affinity under the conditions of manufacture. Hydrated lime as used in the sugar industry consists essentially of calcium hydroxide.

(b) **USE OF HYDRATED LIME.**—Hydrated lime is added to the juice in the dry form, or it is mixed with thin juice or with water to make milk of lime, which is used to precipitate impurities from the juice or sirup.

6. PACKING

(a) Limestone is shipped in bulk in carload lots.

(b) Quicklime is shipped either in bulk in carload lots or in barrels holding 180 pounds net or 280 pounds net each.

(c) Lime powder must be shipped in air-tight containers.

(d) Hydrate lime is generally shipped in paper bags holding 50 pounds net each.

7. MARKING

Each carload of material or fraction thereof shall be legibly marked with the names of the consignor and consignee and with some means of identifying the particular contract on which the shipment is made.

II. REQUIREMENTS

1. COMPOSITION

Lime products shall meet the following requirements as to chemical composition when the sample is taken at the point of delivery and when the result of analysis are calculated to a nonvolatile basis:

	Sugar soluble lime, minimum	Magnesium oxide, maximum	Loss on ignition, maximum
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Limestone ¹ for Steffen process	90	3	-----
Limestone for other purposes	85	3	-----
Quicklime for Steffen process	90	3	2
Quicklime for other purposes	85	3	5
Lime powder	90	3	2
Hydrated lime	86	3	-----

¹ Limestone is calcined before analysis (see Methods of test).

2. SIZE

(a) The size of limestone for shaft kilns shall be a matter of contract between the stone producer and the sugar manufacturer. A tolerance of 2 inches above or below the specified size of stone is allowable.

(b) Lime powder shall be of such size that 98 per cent of it will pass a No. 200 sieve, but not so fine that it will "ball" when rotated in a 40-mesh bolter at 20° C.

(c) Hydrated lime shall be of such size that 98 per cent of it will pass a No. 200 sieve.

III. SAMPLING AND TESTING

1. SAMPLING

(a) BULK SHIPMENTS.—The sample shall be so taken that it will represent an average of all parts of the shipment. One shovelful shall be taken from each 3 tons of material, but not less than 10 shovelfuls per shipment. The sample thus taken shall be broken to 1-inch pieces, mixed, and "quartered" to provide a 15-pound sample for the laboratory.

(b) PACKAGES.—Three per cent of the number of packages shall be selected from different parts of the shipment. A sampling tube shall be used which takes a core of not less than one-half inch in diameter. The tube shall be so inserted into the package that it shall pass substantially the entire length of the package, so as to take a core of the material being sampled from substantially the entire length of the package. Material thus taken from individual packages shall be thoroughly mixed and "quartered," and the sample for testing shall not be less than 15 pounds taken from the material so drawn and prepared.

(c) PRECAUTIONS.—Quicklime, lime powder, and hydrated lime are apt to air-slake. Sampling must, therefore, be conducted as expeditiously as possible in a cool, dry place, and the container for the laboratory sample must be sealed air-tight immediately after it is filled.

2. TESTING

(a) CALCINATION OF LIMESTONE.—Crush the sample to pass a No. 100 sieve. Put approximately 10 g in a platinum crucible and ignite to constant weight at a temperature of 1,000 to 1,100° C., preferably in an electric furnace. The product obtained by this ignition is to be analyzed to determine the chemical composition of the limestone.

(b) SUGAR SOLUBLE LIME.—If the material to be tested is quicklime, grind to pass a No. 100 sieve. Place a 5.000 g sample in a

200 cc flask with 75 to 90 cc of freshly boiled distilled water. Boil gently for 3 minutes. Cool to room temperature. Add 40 to 45 g of commercial white granulated sugar (sucrose) completely dissolved in 40 cc of hot freshly boiled distilled water. Shake vigorously with a rotary motion of the flask, keeping the lime in suspension for 30 minutes. Fill to the mark, mix and filter, rejecting the first runnings. Pipette off 100 cc of the filtrate and titrate with 1.785 normal nitric acid (1 cc=0.050 g CaO), using phenolphthalein as indicator. The cubic centimeters of acid used, multiplied by 2, equals the percentage of sugar soluble lime in the sample as tested. Convert into per cent on nonvolatile basis.

(c) **MAGNESIA.**—The magnesia determination is made after removing silica, iron oxide, alumina, and lime. Ignite 1 g of the sample strongly in a platinum crucible for 15 minutes. Cool, and weigh to obtain weight of nonvolatile sample taken. Moisten the residue and dissolve it in 1:1 hydrochloric acid, evaporate to dryness on the steam bath, take up in 5 cc of hydrochloric acid, add 50 cc of water, and filter off the silica. To the filtrate add 5 cc of hydrochloric acid, then make it slightly alkaline with ammonia, heat to boiling, and filter off the oxides of iron and aluminum. Acidify the filtrate slightly, heat to boiling, add ammonium oxalate slowly to excess, then ammonia slowly until in slight excess. After the precipitate has settled for one to two hours filter and wash with an ammoniacal ammonium oxalate solution. Reserve the filtrate. Ignite the precipitate. When cool moisten it, dissolve in hydrochloric acid, and reprecipitate. Filter off the calcium oxalate, washing with ammoniacal ammonium oxalate solution. Discard the precipitate, combine the filtrate with the first one, slightly acidify with hydrochloric acid, and add an excess of microcosmic salt. Then slowly add ammonia until a crystalline precipitate forms or the solution is alkaline. Finally add one-fifth of the volume of ammonia water in excess. After the precipitate has fully formed, preferably by standing overnight, filter and wash with dilute ammonia water (5 per cent by volume). Dissolve the precipitate in a slight excess of hydrochloric acid, add one or two drops of phosphate solution, and reprecipitate by adding ammonia while stirring, finally adding 2 or 3 cc of ammonia in excess. Filter, wash with 5 per cent ammonia, and ignite to constant weight. Weight of magnesium pyrophosphate $\times 0.3621$ = weight of magnesium oxide.

(d) **LOSS ON IGNITION.**—Ignite 1 g of the sample in a platinum crucible to constant weight at a temperature of 1,000 to 1,100° C.

(e) **FINENESS.**—Weigh out 20 g of lime powder or hydrated lime, transfer to a No. 200 sieve, and brush the material carefully with a camel's-hair brush until practically no more powder passes through.

Transfer the coarse residue to a weighing capsule or watch glass and weigh. Multiply the weight by 5 and deduct from 100 to obtain the percentage passing the No. 200 sieve. If desired, the powder may be spread in a thin layer in the sieve, heated for 15 minutes in an air bath at 100 to 110° C., and while still hot brushed through the sieve as described above. All sieves used in testing shall meet the requirements of the United States Standard Sieve Scale, Bureau of Standards Letter Circular 74.

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